

# Synchrotron Beam Line Operation and Development

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*The Synchrotron Radiation Program at NIST/MSEL includes the development and operation of beam stations at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory, and at the Advanced Photon Source (APS) at Argonne National Laboratory. The emphasis is on microstructure characterization, where NIST scientists, and researchers from industry, universities and other government laboratories perform state-of-the-art measurements on advanced materials.*

The UNICAT collaboration at the Advanced Photon Source, which includes NIST, the University of Illinois, Oak Ridge National Laboratory, and UOP Research Center, includes research techniques that require widely varying characteristics of the incident X-ray beam. To meet the technical demands of our diverse experimental portfolio, the UNICAT beam lines on Sector 33 exploit the brilliance of the APS source to deliver X-rays with high flux and with energy resolution better than or equal to the lifetime-broadened core-hole widths for all accessible K and L shells. The insertion device beam line, 33-ID, became operational November 1, 1999, and as of November 1, 2000, will entertain independent investigators as well as collaborative access team members. The bending magnet beam line, 33-BM, is going into commissioning December 15, 2000 and is expected to become fully operational by the end of 2001.

Ultra-small-angle X-ray scattering (USAXS) is the first of the NIST-responsible experiments to come on line. USAXS provides data in the usually inaccessible Q range (where  $Q = (4\pi/\lambda)\sin\theta$ ,  $\lambda$  is the x-ray wavelength and  $\theta$  is one-half the angle of scatter) down to  $1.5 \times 10^{-4} \text{ \AA}^{-1}$ . In that role, USAXS fills the gap between visible light scattering and pinhole small angle cameras. The UNICAT instrument, which has had nearly a year of full operation, includes several capabilities that make it one of the best in the world. It covers an unprecedented Q range out to  $1 \text{ \AA}^{-1}$ ; it can operate as an "effective pinhole" instrument, where the scattering data is not line-smeared; anomalous-USAXS can be used to uncover and quantify previously inseparable microstructures; and it supports the remarkable new technique of USAXS-imaging. In the past year, the USAXS instrument enjoyed the highest demand of any instrument on UNICAT, and it is the first instrument to be requested by independent investigators.

In the coming year, we are looking forward to commissioning three new NIST-responsible experiments: X-ray topography, X-ray microtomography, and X-ray absorption fine structure. The microtomography is a new capability which is expected to achieve a resolution of  $0.3 \text{ \mu m}$  by a form of in-line near-field holography. This resolution is nearly an order of magnitude better than the current state of the art.

At the NSLS, we operate two beam lines: U7A and X23A2. Numerous improvements have been made to the ultra-soft X-ray (U7A) Materials Science End Station over the past year. Continuous cleaning of the grating optical surfaces became a practical reality with the installation of a pair of special ion pumps and arranging a controlled leak of oxygen gas into the grating chamber. An energy-resolving electron yield detector was installed to enhance and improve the definition of the surface sensitivity of our NEXAFS measurements; this detector is fully operational and is a very significant improvement in sensitivity over the previous design. A new linear drive, that moves 100,000 times faster than our old drive was commissioned and is now in continuous use. In a Phase II SBIR, designs were finalized for the  $I_0$  four slit diode with visible light barrier. In addition, the mechanical setup and actuators for this detector were designed. Finally, the design and construction of ultra high efficiency Si (Li) and high purity Ge energy dispersive detectors with ultra-thin surface dead-layers is underway; its purpose lies in improving our X-ray detection sensitivity at energies below 1 keV. This would be a very significant advance in the quantitative analysis of light elements performed in electron micro probes in many analytical and research laboratories in the United States.

The X23A2 beam line provides a stable, scanning, highly monochromatic X-ray beam in the energy range from 4.9 keV to over 30 keV. The X-ray absorption fine structure (XAFS) spectroscopy done on this line is capable of probing: the short range order in crystalline and amorphous materials; one element at a time, and it provides information on the number, distance and chemical identity of the neighbors of the absorbing atom.

## Contributors and Collaborators

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